DTIC FILE COPY

5471-MS-01

(2)

AD-A199 400

Title of research project : NEW FLUORIDE GLASSES FOR THE PREPARATION OF INFRARED OPTICAL FIBERS

Principal investigator : Professor J. LUCAS, University of Rennes

Contractor : CENTRE REGIONAL ETUDES BRETAGNE SCIENCES, CREBS
PRESIDENCE DE L'UNIVERSITE
Rue du Thabor - 35000 RENNES (France)

Contract number : DAJA 45-86-C-0050

Fourth periodic report : start sept. 1987 ; end : march 1988

The research reported in this document has been made possible through the support and sponsorship of the U.S. Government through its European Research Office of the U.S. Army. ~~This report is intended only for the internal management use of the contractor and the U.S. Government.~~

Title of research project : NEW FLUORIDE GLASSES FOR THE PREPARATION OF INFRARED OPTICAL FIBERS

Principal investigator : Professor J. LUCAS, University of Rennes

Contractor : CENTRE REGIONAL ETUDES BRETAGNE SCIENCES, CREBS
PRESIDENCE DE L'UNIVERSITE
Rue du Thabor - 35000 RENNES (France)

Contract number : DAJA 45-86-C-0050

Fourth periodic report : start sept. 1987 ; end : march 1988

The research reported in this document has been made possible through the support and sponsorship of the U.S. Government through its European Research Office of the U.S. Army. ~~This report is intended only for the internal management use of the contractor and the U.S. Government.~~

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER Fourth	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) STUDY OF NEW FLUORIDE GLASSES FOR THE PREPARATION OF INFRARED OPTICAL FIBERS.		5. TYPE OF REPORT & PERIOD COVERED Interim report - sept. 87/march 88
		6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s) J. LUCAS, Professor		8. CONTRACT OR GRANT NUMBER(s) DAJA 45-86-C-0050
9. PERFORMING ORGANIZATION NAME AND ADDRESS University of Rennes, Chimie Minérale D, Campus de Beaulieu, 35042 Rennes Cédex (France)		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS R & D 5477 MS 01
11. CONTROLLING OFFICE NAME AND ADDRESS CENTRE REGIONAL ETUDES BRETAGNE SCIENCE, Présidence de l'Université, rue du Thabor - 35000 Rennes		12. REPORT DATE
		13. NUMBER OF PAGES
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		15. SECURITY CLASS. (of this report) Non classified
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

Free

This document has been approved for public release and sale; its distribution is unlimited.

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

FLUORIDE GLASSES, INDIUM-BASED, ZIRCONIUM, ~~FREE PREFORM~~ OPTICAL FIBERS, VISCOSITY, PURIFICATION.

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

New zirconium-free fluoride glasses are investigated for I.R. optical fibers operating up to 5-5.5 µm. A five-component glass having the composition in molar percent fluoride $Ba_{30}In_{30}Zn_{20}Y_{10}Th_{10}$ has been optimized for rods and preforms preparation. F/Cl substitution as well as Na adddition allow small variations in the refractive index. Two attempts on rods fibering were positive and demonstrated the possibility of obtaining BIZYT optical fibers without significant crystallization. Purification of indium and Yb based materials has been untertaken.

DD FORM 1 JAN 73 1473 EDITION OF 1 NOV 65 IS OBSOLETE

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

FOURTH PERIODIC REPORT

In the three previous periodic reports, we described a new family of fluoride glasses not containing zirconium either aluminium and having consequently a multiphonon edge shifted towards 8 µm instead of 6.5-7 µm. The expected ultratransparency of such materials was estimated to be around 10^{-3} dB/km at 3 µm with losses calculated to be hundred times lower than for the fluorozirconate glasses in the 5 µm region.

This report will discuss the following points :

1. Viscosity measurement after Tg
2. Fibering conditions of the BIZYT glass : rod and preform
3. Some preliminary characteristics of the first fibers
4. Purification problems associated with In and Yb fluorides.

1. Viscosity measurements

These results have been obtained in collaboration with Professor MOYNIHAN's group at RPI, N.Y. The method of beam bending has been used on a sample of BIZYT glass prepared in dry and controlled conditions.

The following viscosity η in poises has been measured for different temperatures : $\eta = 10^{13}$ at 332° C, $\eta = 10^{11}$ at 354° C and $\eta = 10^{9}$ at 377° C. By extrapolation, $\eta = 10^{6}$ poises is expected at 415° C. Note that the crystallization temperature is 447° C and that the normal viscosity conditions for traditional fluoride glass fibering correspond to 10^{5}, 10^{6} poises.

2. Fiber preparation from BIZYT glass

In the previous reports, we mentionned that the BIZYT glass was the best crystallization resistant Zr-free fluoride glass. The three main obstacles to face with this glass are :

a) avoiding small cristallites formation during preform casting and then fiber drawing

b) combining two glasses with refractive index difference of about 1 % and having the same thermal behaviour

c) combining two glasses having compatible expansion coefficients in order to get core-clad structure without stress and brake during cooling.

the fibering experiments have been made in collaboration with C.G.E. Marcoussis Laboratories and C.N.E.T. Lannion. The rods and preforms were prepared in Rennes and were respectively one rod of BIZYT, one preform with core BIZYT and clad BIZYT + 5 % NaF, one preform with a core BIZYT in which 4 % BaF_2 was substituted by 4 % $BaCl_2$ and a clad of BIZYT.

The rod or preform was 10 cm long with a diameter $\emptyset$ = 0.85 cm. The samples were all surface polished and prepared according to the conditions presented in the third report. The rods and samples were jacketted with PTFE. When rods or preforms are used without protection, crystallization is often observed. The drawing conditions were the following :

a) the preform is maintained in a He atmosphere with a maximum of 6 ppm H_2O

b) the drawing furnace giving a sharp gradient temperature zone is preheated at 384° C

c) the preform is introduced in the heating zone and, after 5-8 min, the extremity of the rod begins to melt and form a drop which falls down in generating the fiber

d) for this temperature, the drawing rate is about 1 m/minute.

e) with the rods only , 40 m of fibers at C.G.E. and 160 m at C.N.E.T. have been drawn at the first experiments.

The figure represents the section of fibers with the PTFE coating ; the diameter is 140 μm.

The Na doped preform has been also successfully fibered while the Cl doped preform showed some crystallization at the interface (note that the preform was already slightly crystallized).

3. Preliminary characterizations of the BIZYT fibers

Some preliminary tests have been undertaken in order to have a rough estimation on those prototype first fibers. The total transmission spectrum shows an almost continuous loss of few dB/m (6-8) from 1.5 to 5 µm indicating that the scattering loss due to crystallites and bubbles is dominant. The tensile strength has been measured on the same fiber and estimated to be about 60 MPa.

It is concluded that this first fiber has a poor optical quality as expected but that the multiphonon losses were effectively shifted towards the 5 µm region.

4. Purification of Yb and In compounds

The most dangerous absorbing impurities associated with Yb^{3+} is Nd^{3+} with an absorption peak at 2.55 µm. Extraction technics using organic esters have been proved to be efficient (Nd^{3+} is non detectable by ICP-plasma).

Indium based materials are purified as $InCl_3$ by sublimation. $InCl_3$ has a high vapor pressure compared to Fe, Ni, Co, Cu chlorides.

This report can be concluded in a positive way because we have demonstrated during the last five months the possibility of fibering the BIZYT glass. The first experiments were very optimistic and we are conscient that the first parameter we have to optimize is the optical quality of the preform.

Our program is now to adjust two glasses with Δn of about 1 % compatible in expansion and resistance to crystallization. Cl/F, Ba/Na, Th/rare-earth substitutions will be tried to reach this goal. We hope to decrease in a near future the scattering loss.

Figure - Section of a BIZYT fluoride fiber, Ø = 140 µm, with a PTFE jacket